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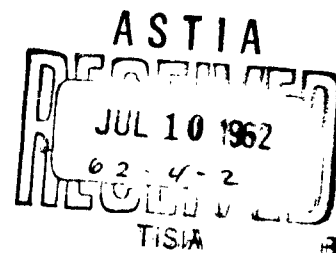
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SUITABILITY OF VITON B O-RINGS FOR USE IN 3000 PSI HYDRAULIC SYSTEMS
CONTAINING PETROLEUM BASE FLUID OR CELLULUBE 220
FINAL REPORT

RUBBER LABORATORY

MARE ISLAND NAVAL SHIPYARD



TECHNICAL REPORT

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SUITABILITY OF VITON B O-RINGS FOR USE IN 3000 PSI HYDRAULIC SYSTEMS
CONTAINING PETROLEUM BASE FLUID OR CELLULUBE 220

FINAL REPORT

Project No. S-F013-13-01

Task No. 907

Identification No. 48-907-2

RUBBER LABORATORY
MARE ISLAND NAVAL SHIPYARD
VALLEJO, CALIFORNIA

Report No. 92-13

Prepared 29 June 1962

REFERENCES

- (a) BUSHIPS ltr F013-13-01 Ser 634C1-694 of 17 Jul 1961
- (b) BUSHIPS ltr F013-13-01 Ser 634C1-1423 of 19 Sep 1960
- (c) Military Specification MIL-H-19457 of 30 Aug 1961; "Hydraulic Fluid, Fire Resistant"
- (d) E. I. du Pont de Nemours & Co., Elastomer Chemicals Dept: report 59-4 of Oct 1959; "Viton B"
- (e) NAVSHIPYD MARE Rubber Laboratory report 92-8 of 10 Mar 1961; "Suitability of Viton B O-rings for Use in 3000 psi Hydraulic Systems Containing Petroleum Base or Phosphate Ester Fluids, Progress Report No. 1"
- (f) NAVSHIPYD MARE Rubber Laboratory report 92-10 of 24 Jul 1961; "Suitability of Viton B O-rings for Use in 3000 psi Hydraulic Systems Containing Petroleum Base or Phosphate Ester Fluids, Progress Report No. 2"
- (g) NAVSHIPYD MARE Rubber Laboratory report 92-11 of 29 Nov 1961; "Suitability of Viton B O-rings for Use in 3000 psi Hydraulic Systems Containing Petroleum Base or Phosphate Ester Fluids, Progress Report No. 3"
- (h) NAVSHIPYD MARE Rubber Laboratory report 92-12 of 20 Feb 1962; "Suitability of Viton B O-rings for Use in 3000 psi Hydraulic Systems Containing Petroleum Base or Phosphate Ester Fluids, Progress Report No. 4"
- (i) BUSHIPS ltr F013-13-01 Ser 634C1-182 of 15 Feb 1962
- (j) Military Specification MIL-P-5516B; "Packings and Gaskets, Preformed, Petroleum and Hydraulic Fluid Resistant"
- (k) NAVSHIPYD MARE ltr F013-13-01 Ser 392-11117 of 26 Mar 1962
- (l) Federal Test Method No. 601 of 12 Apr 1955

INTRODUCTION

1. The Rubber Laboratory was instructed by the Bureau of Ships in references (a) and (b) to develop a simulated service test for evaluating Viton B O-rings for use in 3000 psi hydraulic systems containing either phosphate-ester or petroleum-base fluid. The Laboratory was also instructed to prepare a specification covering Viton B seals of all types intended for service in 3000 psi hydraulic systems containing these fluids.

2. References (a) and (b) identified one of the fluids to be used in this investigation by the generic term "phosphate ester". This implied that any one of several commercial phosphate-ester fluids could be used in the tests. The Laboratory selected Cellulube 220 for this purpose. Cellulube 220 is an aryl phosphate ester manufactured by the Celanese Corporation of America. Cellulube 220 is being used by the Navy in some submarine hydraulic systems, and conforms to specification MIL-H-19457, reference (c).

3. It should be pointed out that Viton B, although compatible with petroleum-base fluids and Cellulube 220, is not compatible with all phosphate-ester fluids. For example, according to reference (d) a Viton B vulcanizate increased in volume by 97% when immersed for 7 days at 300°F in Skydrol 500, another phosphate-ester fluid.

4. ASTM No. 3 oil (ASTM Test Method D471-59T) was the petroleum-base fluid used in these tests in accordance with reference (b).

REVIEW OF EARLIER WORK ON THIS PROJECT

5. As directed in reference (b), the Laboratory developed tests to simulate service conditions of O-rings in submarine hydraulic systems, and subjected

Viton and nitrile rubber O-rings to these tests. In Report 92-8, reference (a), two tests are described. One test evaluated the O-rings for dynamic service. This test utilized a piston with O-ring seals and Teflon backup rings at both ends, which was moved up and down in a cylinder through a stroke of 1.5 inches, 100 times per minute. The test fluid at a pressure of 3000 psig was introduced between the O-rings, and the leakage past the O-rings was collected and measured. The pressure was held for 9 minutes and released for 1 minute periodically.

6. The second test evaluated the O-rings for static service. The O-rings and Teflon backup rings were utilized as static radial seals, with the hydraulic pressure applied between two seals. The pressure of the test fluid was cycled from 0 to 3000 psig and back to 0 psig, 10 times per hour, with a dwell time of 3 minutes at each pressure. Leakage past the seals was measured by noting the loss of fluid from the pump reservoir.

7. The tests simulating static service showed that 70-Shore nitrile rubber O-rings (MIL-P-5516B, Class B) withstood ASTM No. 3 oil satisfactorily, and that 90-Shore Viton B O-rings withstood Cellulube 220 satisfactorily. All of these tests were performed at 300°F, which was much hotter than the maximum oil temperature (ca. 160°F) in submarine hydraulic systems. Viton B O-rings were not tested statically with ASTM No. 3 oil because this rubber is known to be affected less by petroleum-base oils than by Cellulube 220.

8. The tests simulating dynamic service showed that the 70-Shore nitrile rubber O-rings sealed ASTM No. 3 oil at 300°F as long as the Teflon backup rings withstood the treatment. The early failures of the Teflon backup rings at 300°F also limited the serviceability of 70-Shore and 90-Shore Viton B O-rings sealing Cellulube 220. Even when the dynamic test was performed at 180°F, the Teflon backup rings tended to wear excessively.

9. The data on dynamic testing which appeared in Report 92-10, reference (f), confirmed what was found previously. New information was obtained on the ability of 70-Shore Viton B O-rings to withstand ASTM No. 3 oil at 300°F. Here, again, the life of the Teflon backup rings was the limiting factor.
10. Report 92-11, reference (g), described dynamic tests performed at 160°F, which was a more realistic test temperature from the standpoint of actual service. Here it was shown that the 70-Shore Viton B O-rings and 70-Shore nitrile rubber O-rings sealed Cellulube 220 for many kilocycles of piston reciprocation. The Teflon backup rings stood up well at this temperature. The nitrile rubber O-rings were considerably swollen by the Cellulube 220 and badly abraded, but these circumstances did not affect their ability to seal this fluid at 3000 psi pressure under the conditions of this test.
11. Report 92-12, reference (h), described further dynamic testing at 160°F of 70-Shore Viton B O-rings with Teflon backup rings. The fluid sealed was Cellulube 220. These O-rings and backup rings withstood as many as five million cycles of piston reciprocation without excessive leakage. This report also presented the results of proposed specification tests performed on commercial Viton B O-rings (Compound A, Appendix 4) and Viton B O-rings compounded by the Laboratory. These specification tests did not include a simulated service test; this was in accordance with the Bureau's suggestion in reference (i). Among the proposed specification tests was a test for corrosion of metals by the O-rings similar to the test required by specification MIL-P-5516B, reference (j). The ambient air in these tests has high relative humidity. Both types of Viton B O-rings were found to corrode steel and bronze. This finding was a major setback to the progress of the work on this project.
12. The important findings and accomplishments of the previous work on this project are summarized below.

- a. Viton B O-rings with Teflon backup rings were found to seal ASTM No. 3 oil and Cellulube 220 at 3000 psi pressure and 300°F in static service.
- b. Viton B O-rings with Teflon backup rings were found to seal these fluids at 3000 psi pressure and 160°F in dynamic service.
- c. Specification tests for seals intended for service with either of these fluids were proposed. These tests did not include a simulated service test.
- d. The Viton B O-rings were found to corrode steel and bronze after humidification.

EFFECT OF VARIATIONS IN COMPOUNDING ON CORROSION OF METALS

13. The problem yet to be resolved was how to make Viton B O-rings which would not corrode steel or bronze. The first effort to reach this objective consisted of changes in the compounding of the Viton B stock.

14. Viton B contains flourine and, in common with other carbon-hydrogen-halogen polymers, it tends to liberate the halogen acid when heated during vulcanization. This reaction is autocatalytic and can lead to complete decomposition of the polymer if allowed to proceed unhindered. Viton B and the other halogen-containing polymers are usually compounded with a stabilizer and/or acid acceptor to stop this reaction by removing any halogen acid which has been liberated. The Laboratory-compounded Viton B stock, 377-108, recommended in Report 92-12 for the manufacture of O-rings, contained magnesium oxide (Maglite D) for this purpose.

15. O-rings made from 377-108 corroded steel and bronze after the O-rings had been exposed to humid air for 3 days (MTL-P-5516B test). It appeared that residual acid may have been present in the vulcanizate. Accordingly, an extensive compounding survey was made wherein the effect of the following variables

were studied in an effort to obtain a vulcanizate without residual acid. The formulations of the stocks studied are given in Appendix 1.

- a. Litharge, red lead, lead dioxide, and dibasic lead phosphite (Dyphos) were substituted individually for the magnesium oxide in 377-108. Lead compounds are commonly used stabilizers for halogen-containing polymers.
- b. Red lead, lead dioxide and calcium oxide were added individually in addition to magnesium oxide.
- c. Calcium carbonate (Purecal U), which reacts with acids, was substituted for carbon black (Thermax) as the reinforcing filler.
- d. Fine silica (Silstone 120) was substituted for carbon black, since the black may have been adsorbing the halogen acid from the polymer and liberating it when exposed to the humid atmosphere.
- e. Peroxide curing agents (DiCup 40C, Varox) were tried in lieu of the diamine curing agent (Diak No. 3) since it was thought that the latter might be catalyzing the decomposition of the polymer.

16. In addition to the compounding changes intended to stabilize the Viton B and to remove any liberated acid, stocks containing metal passivators were prepared. The passivators were sodium chromate, zinc chromate and lead chromate. It was hoped that the presence of these passivators in the rubber would protect the metal from corrosion.

17. The formulations, curing times and physical properties of the experimental vulcanizates are given in Appendix 1. All but one of the stocks containing peroxides did not cure.

18. O-rings conforming to the dimensions of AN6230-7 were prepared from each stock which would cure. The tendency of these O-rings to corrode metals was determined according to the procedure given below. This procedure is essentially the same as that given in specification MIL-P-5516B.

The O-rings were washed with Standard 200, an aliphatic solvent of moderate volatility, and placed in a chamber at 100% relative humidity for 72 hours at 74°F. The O-rings were then dipped in Cellulube 220 and clamped between polished metal test plates having a surface roughness of not more than 15 microinches. The metal plates were washed with Standard 200 immediately before use. The clamping force was 20 pounds. The assembly was maintained at 100% relative humidity and 74°F for 4 days. The plates conformed to the following specifications:

Aluminum alloy	Spec. QQ-A-318	Temper H32
Brass	Spec. MIL-B-994	Composition A
Bronze	Spec. QQ-P-330	Composition A
Steel	Spec. MIL-S-6758	Condition C2

19. The results of the corrosion tests are given in Appendix 1, together with physical properties of the stocks tested. When the test plates were separated from the O-rings at the end of the 4-day period, it was found that every vulcanizate tested rusted the steel plates and discolored the bronze plates. Photographs showing typical examples of the corrosion of steel and bronze were included in Report 92-12. The aluminum and brass plates were unaffected. Apparently none of the variations in compounding which were tried had any effect on the formation or presence of corrosive material in the Viton vulcanizates.

20. Early in the investigation one test showed that O-rings of stocks 377-133 and 377-135, containing red lead and lead peroxide, respectively, as well as magnesium oxide, did not attack steel or bronze, and this information was so reported to the Bureau in reference (k). However, when confirmatory tests were run, O-rings of both stocks rusted steel and discolored bronze as badly as the rest of the stocks studied.

EFFECT OF LEACHING WITH WATER ON CORROSION OF METALS

21. In an attempt to remove the corrosive material from Viton B vulcanizates, O-rings of several stocks listed in Appendix 1 and representing the major compounding variations, were leached in boiling distilled water for periods ranging from 6 to 40 hours. The O-rings were then conditioned at 74°F and 100% relative humidity for 72 hours, dipped in Cellulube 220, and clamped between polished test plates for 4 days at 74°F and 100% relative humidity as before. When the plates were examined at the end of the test, it was found that all of the O-rings rusted steel and stained bronze. If anything, a longer period of leaching increased the amount of corrosion. It was concluded that leaching was of no benefit whatever.

EFFECT OF WASHING WITH SOAP AND WATER ON CORROSION OF METALS

22. O-rings of seven different stocks were prepared conforming to AN6230-7. The formulations and curing information for each stock are given in Appendix 2. One set of O-rings of each stock was washed with soap and water, humidified at 100% relative humidity for 72 hours, dipped in Cellulube 220, and clamped for 4 days between polished metal plates conforming to the requirements listed in paragraph 18. A second set of O-rings was treated in the same way except that the O-rings were washed after humidification and just before being dipped in Cellulube 220 and clamped between the test plates. After 4 days at 100% relative humidity, the surfaces of the plates contacted by the O-rings were examined for corrosion.

23. The results of the tests are given in Appendix 2. When the O-rings were washed prior to humidification, all O-rings rusted steel and stained bronze. When washing was done after humidification, none of the O-rings affected steel or bronze. Aluminum and brass plates were not affected by either lot of O-rings. As was noted previously in Appendix 1, O-rings of these same stocks all corroded bronze and steel when the O-rings were washed only in Standard 200 prior to humidification.

24. The results of these tests gave definite evidence that some corrosive material exuded to the surface of the O-rings under conditions of 100% relative humidity, and that corrosion could be eliminated by removing this material with soap and water.

25. In view of the beneficial effect of washing O-rings on the prevention of this corrosion of metal, it was decided to study various washing procedures in order to select the best and most practical one. The various procedures employed are listed in Appendix 3. In order to determine conclusively whether or not the presence of lead oxide in a stock is required to prevent metal corrosion, two stocks were tested. One contained red lead in addition to magnesium oxide and one contained only magnesium oxide. The formulations of the stocks are given in Appendix 3 together with the results of the corrosion tests.

26. From these data it was determined that there was no advantage to adding red lead to Viton stocks. Two washing procedures for O-rings eliminated corrosion: (1) washing with soap and water plus washing with Standard 200 before humidifying, and (2) washing with soap and water after humidification. This latter procedure is recommended since it eliminates use of an inflammable solvent. In practice this procedure would consist of washing Viton O-rings with soap and water just before installation in the equipment in which they are to be used.

27. Stock 377-154 was selected for further investigation because of its excellent properties (Appendix 1) and because it did not corrode the metal test plates when washed as recommended in the preceding paragraph. This stock differs from stock 377-108 previously recommended only in not containing di-isooctyl sebacate as a processing aid. It is the Laboratory's experience that di-isooctyl sebacate is not necessary to obtain satisfactory processing of 70-Shore Viton B stocks. If a manufacturer desires to add di-isooctyl sebacate to the stock to facilitate

processing on large-size equipment, no significant change in physical properties would result as can be seen by comparing the physical properties of stocks 377-108 and 377-154 in Appendix 1. It might, however, become necessary to make special oversize molds, since volatilization of the di-isooctyl sebacate during the oven cure would cause greater shrinkage.

PROPOSED SPECIFICATION REQUIREMENTS FOR VITON B SEALS

28. The purpose of specification tests is to insure that items procured under the specification are suitable for intended use. Usually the test limits are set on the basis of test results on materials which have been shown to be satisfactory in service or in simulated service tests. The selection of the tests depends on the application and is designed to measure those properties essential to satisfactory functioning of the material.

29. The properties important in hydraulic seals are adequate strength to resist tearing and gouging, sufficient hardness to prevent extrusion, and low permanent set so that the O-ring or other seal will fit into its groove after being stretched during installation. The seal must resist swelling by hydraulic fluids, and must not suffer an excessive loss of the above mentioned properties due to fluid immersion. The seal must retain adequate properties after air aging and must not exhibit so much compression set as to prevent proper sealing. The seal must not corrode metals with which it comes in contact.

30. Specimens of stock 377-154 (paragraph 27) and Compound A, a commercial O-ring composition identified in Appendix 4, were tested for the properties listed in the preceding paragraph. The recipe and curing procedure for stock 377-154 are given in Appendix 3.

31. The properties determined and the methods used are listed below. All tests were performed on AN6230-7 O-rings except as noted.

Initial Properties

Tensile strength
Ultimate elongation
Tensile stress (100% elongation)

ASTM Test Method D1414-56T. Six specimens were tested.

Hardness

Method 3021 of reference (1). Readings were taken 15 sec. after application of indenter. Specimen was 1/4 inch thick slab. Four measurements were made.

Permanent set

O-rings were stretched over a cone to 50% elongation, held 10 min., released 10 min., and set was measured as % increase in circumference. Three O-rings were tested.

Specific gravity

Method 14011 of reference (1).

After aging 70 hours at 212°F in air

Method 7221 of reference (1).

Tensile strength
Ultimate elongation
Tensile stress (100% elongation)
Hardness
Compression set

Same as initial measurement.
Same as initial measurement.
Same as initial measurement.
Same as initial measurement.
Method 3311 of reference (1).
Specimens were two 1/4-inch thick x 1.129 inch diameter discs plied to 1/2 inch thickness. Three specimens were tested. Recovery period was 30 min. at 212°F plus 30 min at 74°F.

After aging 70 hours at 212°F in Cellulube 220

Tensile strength
Ultimate elongation
Tensile stress (100% elongation)
Swell

Method 6111 of reference (1) and ASTM Test Method D1414-56T, six specimens were tested.
Method 6211 of reference (1). Tensile strength and tensile stress were based on swollen cross-sectional diameter measured just prior to testing using four specimens.
Same as initial measurement.

Hardness

After aging 70 hours at 212°F in ASTM Oil No. 3

Same properties were measured and same methods were used as with Cellulube 220 above.

After 96 hours at 74°F in 100% relative humidity

Corrosion and adhesion

Paragraph 4.5.6 of Spec MIL-P-5516B,
reference (j) using metals as follows:

Steel	Spec MIL-S-6758 Condition C2
Brass	Spec MIL-B-994, Composition A
Bronze	Spec QQ-P-330 Composition A
Aluminum	Spec QQ-A-318, Temper H32

The fluids used were Cellulube 220 oil
No. 3. The O-rings were washed in soap
and water after humidification and prior
to being wet with the test fluid and
clamped between the test plates.

32. In addition to the above tests and as verification of the value of the washing procedure used, O-rings of stock 377-154 were subjected to the corrosion test above except that the time the O-rings were clamped between the test plates was increased from 96 hours (4 days) to 30 days.

33. The results of the tests and the proposed specification requirements are given in Appendix 5. The data show that the O-rings made from stock 377-154 and Compound A had similar properties. The O-rings of both stocks were judged suitable for use as static or dynamic seals for petroleum-base fluid or Cellulube 220. It is noteworthy that O-rings of stock 377-154 which had been washed with soap and water after humidification did not corrode or even stain metals after 30-days contact.

CONCLUSIONS

34. The results of the foregoing tests lead to the following conclusions:

- a. Stock 377-154 and Compound A are suitable for use in seals for 3000 psi hydraulic systems containing either Cellulube 220 or petroleum-base fluids.

- b. Viton seals should be washed with soap and water just prior to being installed in the equipment in which they are to be used. This treatment will prevent corrosion of the surfaces contacted by the seals.

PROPOSED SPECIFICATION

35. A proposed specification for procurement of seals and gaskets to be used in 3000 psi hydraulic systems containing either petroleum-base or phosphate-ester fluids is given in Appendix 6. This specification was requested by reference (a).

PERSONNEL

Bureau of Ships Project Engineer	W. S. Bourn
Tests performed by	R. D. Ford, Technologist
Supervised by	A. E. Barrett, Supervisory Technologist
Report prepared by	R. D. Ford, Technologist R. E. Morris, Head, Rubber Laboratory

Approved by

R E Morris

R. E. Morris, Head, Rubber Laboratory

APPENDICES

1. Table. Stocks investigated for corrosion and results of tests.
 2. Table. Effect of washing O-rings with soap and water on their tendency to corrode metals.
 3. Table. Effect of various procedures for washing O-rings on their tendency to corrode metals.
 4. Identification of Compound A
 5. Table. Physical properties of stock 377-154 and Compound A and proposed specification requirements
 6. Proposed specification for seals and gaskets to be used in 3000 psi hydraulic systems containing either petroleum-base or phosphate-ester fluids
- Abstract card
- Distribution of report

STOCKS INVESTIGATED FOR CORROSION AND RESULTS OF TESTS
(Sheet 1 of 3)

<u>Stock No. 377-</u>	<u>108</u>	<u>154</u>	<u>109</u>	<u>131</u>	<u>132</u>	<u>133</u>	<u>147</u>	<u>148</u>	<u>149</u>	<u>150</u>	<u>154</u>	<u>157</u>	<u>158</u>
Viton B	100	100	100	100	100	100	100	100	100	100	100	100	100
Thermax	20	20	20	20	20	20	20	20	20	20	20	20	20
Maglite D	15	15	-	-	15	15	30	15	7.5	15	-	-	10
Litharge	-	-	15	-	-	-	-	-	-	-	-	-	-
Red lead.	-	-	-	15	30	15	15	30	15	7.5	-	-	-
Lead dioxide	-	-	-	-	-	-	-	-	-	-	-	-	-
Dyphos	-	-	-	-	-	-	-	-	-	-	-	-	-
Protox 166	-	-	-	-	-	-	-	-	-	-	-	-	-
Di-isoctyl sebacate	3	-	3	-	-	-	-	-	-	-	-	-	-
Stearic acid	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Diak No. 3	3	3	3	4	4	4	3	3	3	3	4	3	3

Cure: 30 min. at 300°F in press plus 1 hr. at 200, 250, 300, 350°F in oven plus 24 hrs. at 400°F in oven.

Tensile strength, psi ⁽¹⁾	2180	2050	1710	1710	1720	2150	2130	2010	2180	2360	1980	2140	1840	1900	2080
Ultimate elongation, %	260	240	310	320	270	190	150	160	280	260	360	200	370	380	230
Tensile stress at 100% elongation, psi	560	570	420	370	500	860	1240	1030	590	700	320	840	420	390	580
Shore A hardness (15 sec) ⁽²⁾	68	70	-	66	70	75	81	75	70	72	-	73	67	65	70
Compression set (72 hrs. at 212°F) ⁽³⁾	-	19	-	25	24	17	28	22	18	17	-	27	26	28	18

Corrosion after 96 hrs. (4)

in contact with:

Steel	All stocks caused rusting of the steel.
Bronze	All stocks caused discoloration of the bronze
Brass	None of the stocks corroded or stained brass.
Aluminum	None of the stocks corroded or stained aluminum.

STOCKS INVESTIGATED FOR CORROSION AND RESULTS OF TESTS
(Sheet 2 of 3)

Stock No. 377-	127	128	129	139	115	116	120	121	122	123	110	111
Viton B	100	100	100	-	100	100	100	100	100	100	100	100
Viton A-HV	-	-	-	100	-	-	-	-	-	-	-	-
Thermax	20	20	20	-	20	20	20	-	-	-	20	20
Maglite D	15	15	15	15	-	-	-	15	15	15	15	-
Sodium chromate	5	-	-	-	-	-	-	-	-	-	-	-
Lead chromate	-	5	-	-	-	-	-	-	-	-	-	-
Zinc chromate	-	-	5	-	-	-	-	-	-	-	-	-
Silatone 120	-	-	-	20	-	-	-	-	20	20	-	-
Litharge	-	-	-	-	15	15	15	-	-	-	-	15
Blanc fixe	-	-	-	-	-	-	-	45	-	-	-	-
Stearic acid	0.5	0.5	0.5	-	0.5	0.5	0.5	-	-	-	0.5	0.5
Di-isooctyl sebacate	-	-	-	-	3	3	3	-	-	-	3	3
Diak No. 3	3	3	3	-	-	-	-	-	-	-	-	-
DiCup 40C	-	-	-	5	-	5	15	5	5	5	5	5
Varox	-	-	-	-	5	-	-	-	-	-	-	-
Copper Inhibitor 65	-	-	-	0.5	-	-	-	-	-	0.5	0.5	0.5

Cure: 30 min. at 300°F in press plus 1 hour at 200, 250, 300, and 350°F in oven plus 24 hrs. in oven at 400°F

Tensile strength, psi ⁽¹⁾	2350	2530	2320	1410	(6)	(6)	(6)	(6)	(6)	(6)	(6)	(6)
Ultimate elongation, %	270	330	310	420								
Tensile stress at 100% elongation, psi	640	500	510	330								
Shore A hardness (15 sec) ⁽²⁾	70	68	70	-	(5)							
Compression set (72 hrs. at 212°F), % ⁽³⁾	19	17	17	-	(5)							

Corrosion after 96 hrs. ⁽⁴⁾

in contact with:

Steel
Bronze
Brass
Aluminum

All stocks caused rusting of steel.
All stocks caused discoloration of bronze.
None of the stocks corroded or stained brass.
None of the stocks corroded or stained aluminum.

STOCKS INVESTIGATED FOR CORROSION AND RESULTS OF TESTS
(Sheet 3 of 3)

<u>Stock No. 377-</u>	<u>151</u>	<u>152</u>	<u>153</u>	<u>130</u>	<u>113</u>	<u>114</u>	
Viton B	100	100	100	100	100	100	
Purecal U	25	25	25	-	-	-	
Thermax	-	-	-	20	20	20	
Meglite D	15	-	15	-	15	15	
Red lead	-	15	15	-	-	-	
Calcium oxide	-	-	-	11	-	-	
Copper (powdered)	-	-	-	-	5	-	
Cuprous oxide	-	-	-	-	-	5	
Di-isooctyl sebacate	-	-	-	-	3	3	
Stearic acid	0.5	0.5	0.5	0.5	0.5	0.5	
Disk No. 3	4	4	4	1.5	3	3	
Cure: 30 min. at 300°F in press plus 1 hr. at 200, 250, 300, and 350°F in oven plus 24 hrs. at 400°F in oven							
Tensile strength, psi (1)	2170	1940	2180	1680	-	-	
Ultimate elongation, %	170	200	160	310	-	-	
Tensile stress at 100% elongation, psi	760	650	1060	410	-	-	
Shore A hardness (15 sec) (2)	70	-	(5) 73	-	-	-	
Compression set (72 hrs. at 212°F), %	12	-	(5) 15	-	-	-	

Corrosion after 96 hrs. (4)
in contact with:

Steel	All stocks caused rusting of the steel.
Bronze	All stocks caused discoloration of the bronze.
Brass	None of the stocks corroded or stained brass.
Aluminum	None of the stocks corroded or stained aluminum.

NOTES:

- (1) Tensile properties measured on AN6230-7 O-rings.
- (2) Hardness measured on 1/4 inch slab.
- (3) Compression set measured on two 1/4 inch x 1.129 inch disks piled together. Set was measured after recovery for 30 min. at 212°F plus 30 min. at 74°F.
- (4) AN6230-7 O-rings were humidified for 72 hours at 100% relative humidity, washed in Standard 200 solvent, dipped in Cellulube 220 and clamped between polished metal plates for 96 hrs. at 100% relative humidity.
- (5) Specimens for these tests became spongy during the oven cure because of low state of cure after the press cure.
- (6) Stock did not cure.

EFFECT OF WASHING O-RINGS WITH SOAP AND WATER ON THEIR TENDENCY TO CORRODE METALS

Stock No. 377-	147	148	149	150	151	152	153
Viton B	100	100	100	100	100	100	100
Thermax	20	20	20	20	20	20	20
Purecal U	-	-	-	-	25	25	25
Red lead	15	30	15	7.5	-	15	15
Maglite D	30	15	7.5	15	15	-	15
Stearic acid	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Diak No. 3	3	3	3	3	4	4	4

Cure: 30 min. at 300°F in press plus 1 hr. at 200, 250, 300 and 350°F in oven plus 24 hrs. at 400°F in oven

Corrosion

O-rings washed 72 hrs. (1)

prior to test

	Rusted	Discolored	Rusted	Discolored	Rusted	Discolored	Rusted	Discolored
Steel	None	None	None	None	None	None	None	None
Bronze	None	None	None	None	None	None	None	None
Brass	None	None	None	None	None	None	None	None
Aluminum	None	None	None	None	None	None	None	None

O-rings washed immediately (2)

prior to test

	Rusted	Discolored	Rusted	Discolored	Rusted	Discolored	Rusted	Discolored
Steel	None	None	None	None	None	None	None	None
Bronze	None	None	None	None	None	None	None	None
Brass	None	None	None	None	None	None	None	None
Aluminum	None	None	None	None	None	None	None	None

NOTES: (1) AN6230-7 O-rings were washed in soap and water. The O-rings were then humidified for 72 hrs at 100% relative humidity, dipped in Cellulube 220, and clamped between polished metal test plates.

(2) AN6230-7 O-rings were humidified for 72 hrs. at 100% relative humidity. They were then washed in soap and water, dipped in Cellulube 220, and clamped between polished metal plates.

**EFFECT OF VARIOUS PROCEDURES FOR WASHING O-RINGS
ON THEIR TENDENCY TO CORRODE METALS**

<u>Stocks</u>	<u>377-150</u>	<u>377-154</u>
Viton B	100	100
Thermax	20	20
Maglite D	15	15
Red lead	7.5	
Stearic Acid	0.5	0.5
Diak No. 3	3	3

Cure: 30 min. at 300°F in press plus 1 hr. at 200, 250, 300 and 350°F in oven plus 24 hrs. at 400°F in oven.

Corrosion of metals by O-rings (1)

O-rings not washed at any time	Rusted steel, discolored bronze	Rusted steel, discolored bronze
O-rings washed with soap and water before humidifying	None	1 of 4 steel plates rusted slightly
O-rings washed with soap and water after humidifying	None	None
O-rings washed with Standard 200 solvent before humidifying	Slight rust on steel, bronze discolored	Slight rust on steel, bronze discolored
O-rings washed with Standard 200 solvent after humidifying	Slight rust on steel, bronze discolored	Slight rust on steel, bronze discolored
O-rings washed with soap and water, then with Standard 200 solvent before humidifying	None	None

(1) All O-rings were AN6230-7 and were humidified 72 hrs. at 100% relative humidity. The O-rings were dipped in Cellulube 220 and clamped between polished metal plates for 96 hrs. at 100% relative humidity.

**PHYSICAL PROPERTIES OF STOCK 377-154 AND COMPOUND A
AND PROPOSED SPECIFICATION REQUIREMENTS**
(Sheet 1 of 2)

<u>TESTS</u> (1)	<u>COMPOUND A</u> <u>(VITON B)</u>	<u>STOCK 377-154</u> <u>(VITON B)</u>	<u>PROPOSED</u> <u>SPECIFICATION</u> <u>REQUIREMENTS</u>
<u>Initial properties</u>			
Tensile strength, psi	2190	2050	Min. 1600
Ultimate elongation, %	210	240	Min. 150
Tensile stress at 100% elongation, psi	860	570	
Shore A hardness (15 second), units	74	70	70 ± 5
Specific gravity	1.91	1.92	
Permanent set, %	1	3	Max. 5
<u>After 70 hrs. at 212°F in air</u>			
Tensile strength, psi	2170	2120	
Tensile strength, retention, %	91	103	Min. 85
Ultimate elongation, %	200	240	
Ultimate elongation, retention, %	95	100	Min. 85
Tensile stress at 100% elongation, psi	940	580	
Tensile stress, retention, %	109	102	
Shore A hardness (15 second), units (2)	69	71	70 ± 5
Change in Shore A hardness, units	-5	+1	
Compression set, % (3)	12	19	Max. 25
<u>After 70 hrs. at 212°F in Cellulube 220</u>			
Tensile strength, psi	1810	1970	
Tensile strength, retention, %	83	96	Min. 80
Ultimate elongation, %	200	280	
Ultimate elongation, retention, %	95	117	Min. 80
Tensile stress at 100% elongation, psi	730	500	
Tensile stress, retention, %	85	88	
Volume change, %	+5	+5	Max. +8, Min. 0
Shore A hardness (15 second), units (2)	66	66	65 ± 5
Change in Shore A hardness, units	-8	-4	

(1) All properties measured on AN6230-7 O-rings except as noted.

(2) Measured on 1/4-inch thick molded sheet.

(3) Measured on two 1/4-inch thick x 1.129-inch diameter disks plied to form a 1/2-inch thick specimen.

**PHYSICAL PROPERTIES OF STOCK 377-154 AND COMPOUND A
AND PROPOSED SPECIFICATION REQUIREMENTS
(Sheet 2 of 2)**

<u>TESTS</u> (1)	<u>COMPOUND A</u> <u>(VITON B)</u>	<u>STOCK 377-154</u> <u>(VITON B)</u>	<u>PROPOSED</u> <u>SPECIFICATION</u> <u>REQUIREMENTS</u>
<u>After 70 hrs. at 212°F in ASTM Oil No. 3</u>			
Tensile strength, psi	2040	2010	
Tensile strength, retention, %	93	98	Min. 85
Ultimate elongation, %	220	250	
Ultimate elongation, retention, %	95	96	Min. 85
Tensile stress at 100% elongation, psi	780	560	
Tensile stress, retention, %	91	98	
Volume change, %	+2	+2	Max. +5; Min. 0
Shore A hardness (15 second), units (2)	69	68	70±5
Change in Shore A hardness, units	-5	-2	
<u>Corrosion and adhesion after 96 hours at 100% relative humidity, specimen wet with Cellulube 220</u>			
Steel	None	None (4)	None (5)
Bronze	None	None	None
Brass	None	None	None
Aluminum	None	None	None
<u>Corrosion and adhesion after 96 hours at 100% relative humidity, specimen wet with ASTM Oil No. 3</u>			
Steel	None	None	None (5)
Bronze	None	None	None
Brass	None	None	None
Aluminum	None	None	None

- (1) All properties measured on AN6230-7 O-rings except as noted.
 (2) Measured on 1/4-inch thick molded sheet.
 (4) When Stock 377-154 O-rings were clamped for 30 days no corrosion, staining or adhesion occurred on any of the metals.
 (5) Discoloration or staining shall not be considered detrimental if the metal surface is not roughened.

PROPOSED SPECIFICATION FOR

SEALS AND GASKETS TO BE USED IN 3000 PSI HYDRAULIC SYSTEMS CONTAINING EITHER PETROLEUM-BASE OR PHOSPHATE-ESTER FLUIDS

1. SCOPE

1.1 This specification covers packings and gaskets intended for use in 3000 psi hydraulic systems containing either a petroleum-base fluid or a phosphate-ester fluid conforming to Military Specification MIL-H-19457.

2. APPLICABLE DOCUMENTS

2.1 The following documents, of the issue in effect on date of invitation for bids, form a part of this specification.

SPECIFICATIONS

FEDERAL

QQ-A-318	Aluminum Alloy, Plate and Sheet
QQ-P-330	Phosphor Bronze Bars, Plates, Rods, Sheets, Strips, Flat Wire, and Structural and Special Shaped Sections

MILITARY

MIL-B-994	Brass, Naval, Rods, Wire, Shapes, Forgings, and Flat Products (Flat Wire, Strip Sheet, Bar and Plate)
MIL-S-6758	Steel, Chrome-Molybdenum (4130) Bars and Reforging Stock (Aircraft Quality)
MIL-H-19457	Hydraulic Fluid, Fire Resistant

STANDARDS

FEDERAL

FED-STD-601	Rubber: Sampling and Testing
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MILITARY

MS28772	Packing, D-Ring, Shock Strut
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AIR FORCE-NAVY AERONAUTICAL

AN6225	Packing: V-Ring Hydraulic
AN6226	Packing: U-Cup Hydraulic
AN6227	Packing: O-Ring Hydraulic
AN6230	Gasket: O-Ring Hydraulic

3. REQUIREMENTS

3.1 Materials. The items covered by this specification shall be made from compounds which have been tested as specified herein, and which are compatible with Medium No. 3 of Method 8001, FED-STD-801, and with fluids conforming to MIL-H-19457.

3.2 Design and construction.

3.2.1 Shape and dimensions. The shape and dimensions of D-ring packings shall conform to MS28772, V-ring packings to AN6225, U-cup packings to AN6226, O-ring packings to AN6227, and O-ring gaskets to AN6230.

3.2.2 Finish. Mold flash shall be removed from the packings and gaskets in such a manner that they conform to the requirements specified herein and on the applicable standards. Any method used to trim the mold flash on O-rings shall not remove the original mold finish of the ring over an area wider than 1/16-inch maximum adjacent to the flash.

3.2.2.1 The sealing surfaces of packings and gaskets shall not have defects such as pits, raised spots, tool marks resulting from mold imperfections, which are greater than 0.003 inch in height or depth. Such imperfections shall not be grouped closely, and shall cover not more than 10% of the surface. The entire surface of O-ring packings and gaskets shall be considered sealing surface.

3.2.2.2 The non-sealing surface of U-cup and V-ring packings shall be free from irregularities greater than ± 0.010 inch, and shall contain no cuts, laps, cracks, seams, or other defects. The irregularities present shall not cause the packings to exceed the stack height tolerances.

3.3 Properties of rubber.

3.3.1 Initial properties. When tested as specified in 4.2.1, the rubber shall have the following properties:

Tensile strength, minimum, psi	1600
Ultimate elongation, minimum, psi	150
Hardness, Shore A durometer	70 \pm 5
Permanent set, maximum, %	5

3.3.2 Oven Aging. When subjected to the oven aging test specified in 4.2.2, the rubber shall have the following properties:

Tensile strength, minimum retention, %	85
Ultimate elongation, minimum retention, %	85
Hardness, Shore A durometer	70 \pm 5
Compression set, maximum, %	25

3.3.3 Aging in MIL-H-19457 Fluid. After immersion in fluid conforming to MIL-H-19457, as specified in 4.2.3, the rubber shall have the following properties:

Tensile strength, minimum retention, %	80
Ultimate elongation, minimum retention, %	80
Hardness, Shore A durometer	65 \pm 5
Change in volume, maximum, %	+8
Change in volume, minimum, %	0

3.3.4 Aging in Medium No. 3. After immersion in Medium No. 3, as specified in 4.2.3, the rubber shall have the following properties:

Tensile strength, minimum retention, %	85
Ultimate elongation, minimum retention, %	85
Hardness, Shore A durometer	70 ₋₅
Change in volume, maximum, %	+5
Change in volume, minimum, %	0

3.3.5 Corrosion and adhesion. When subjected to the corrosion test described in 4.2.4, the O-rings shall not corrode the specified metals sufficiently to roughen the metal surface. Staining or discoloration of the metal is permissible if the surface is not roughened. The O-rings shall not adhere to the metal surfaces.

4. QUALITY ASSURANCE PROVISIONS

4.1 Acceptance tests. Tests shall be conducted at a laboratory satisfactory to the Bureau of Ships. Tests shall measure the physical properties listed in 3.3 using the test procedures described in 4.2.

4.1.1 Sampling instructions. The acceptance test samples shall consist of the following:

- 43 AN6230-7 O-rings.
- 6 Compression set specimens 1/4-inch thick by 1.129 inches in diameter.
- 6 Hardness specimens 1/4-inch thick by 2 inches in diameter.

4.1.1.1 O-rings, compression set specimens, and hardness specimens shall have the same compounding and cure as the accompanying gaskets or packings.

4.2 Test procedures.

4.2.1 Initial properties

4.2.1.1 Tensile strength. The tensile strength shall be determined by Method 4111 of FED-STD-601 except that specimens shall consist of six AN6230-7 O-rings. The mean value shall be reported.

4.2.1.2 Ultimate elongation. The ultimate elongation shall be determined by Method 4121 of FED-STD-601 on the same specimens used in 4.2.1.1. The mean value shall be reported.

4.2.1.3 Hardness. Hardness shall be measured with a Shore A durometer on molded specimens 1/4-inch thick by 2 inches in diameter, in accordance with Method 3021 of FED-STD-601 except that the hardness reading shall be taken 15 seconds after the indenter of the durometer is pressed against the specimen.

4.2.1.4 Permanent set. Permanent set shall be measured on three AN6230-7 O-rings at room temperature (75°F±15°F). The ID of the O-rings shall be measured. The O-rings shall then be stretched over a cone until the ID of the O-ring has been stretched 50% (1.5 times its original value). After 10 minutes the O-rings shall be removed and allowed to rest for 10 minutes, and the new ID measured. Permanent set shall be calculated as the percentage increase in ID. The mean value shall be reported.

4.2.2 Properties after aging in air.

4.2.2.1 Tensile strength and ultimate elongation. Tensile strength and ultimate elongation shall be determined on six AN6230-7 O-rings by Method 7221 of FED-STD-601 except the aging period shall be 70 hours at $212^{\circ}\pm 2^{\circ}\text{F}$. The mean value shall be calculated and used in computing the percent retention of these properties after aging.

4.2.2.2 Hardness. Hardness shall be measured as in 4.2.1.3 on specimens which have been aged for 70 hours at $212^{\circ}\pm 2^{\circ}\text{F}$. The specimens shall be allowed to recover at room temperature ($75^{\circ}\pm 5^{\circ}\text{F}$) for not less than 16 nor more than 96 hours before measuring the hardness.

4.2.2.3 Compression set. Compression set shall be measured according to Method 3311 of FED-STD-601 except that three specimens shall be tested, each consisting of two molded disks 1/4-inch thick by 1.129 inches in diameter plied together. The aging period shall be 70 hours at $212^{\circ}\pm 2^{\circ}\text{F}$. At the end of the aging period, the specimens shall be removed from the compression apparatus and allowed to recover for 30 minutes in an oven at $212^{\circ}\pm 2^{\circ}\text{F}$. The specimens shall then be removed from the oven and allowed to recover for an additional 30 minutes at room temperature ($75^{\circ}\pm 5^{\circ}\text{F}$). The final thickness shall then be measured.

4.2.3 Properties after aging in MIL-H-19457 fluid or in FED-STD-601 Medium No. 3 test fluid.

4.2.3.1 Tensile strength and ultimate elongation. Tensile strength and ultimate elongation shall be measured on six AN6230-7 O-rings by Method 6111 of FED-STD-601. Tensile strength shall be based on the swollen cross-sectional diameter measured immediately before testing. The immersion period shall be 70 hours at $212^{\circ}\pm 2^{\circ}\text{F}$. The mean values shall be calculated for use in computing the percent retention of these properties after the aging.

4.2.3.2. Hardness. Hardness shall be measured as in 4.2.1.3 on specimens which have been immersed in the test fluid for 70 hours at $212^{\circ}\pm 2^{\circ}\text{F}$, then removed and cooled in a container of the same fluid at room temperature ($75^{\circ}\pm 5^{\circ}\text{F}$) for 30 ± 5 minutes. The specimens shall be removed, dipped momentarily in acetone, blotted dry, and the hardness measured.

4.2.3.3 Change in volume. Change in volume after liquid immersion shall be measured on three AN6230-7 O-rings by Method 6211 of FED-STD-601. The immersion period shall be 70 hours at $212^{\circ}\pm 2^{\circ}\text{F}$.

4.2.4 Corrosion and adhesion. Eight AN6230-7 O-rings shall be prepared for corrosion testing by conditioning them in a chamber maintained at 92% minimum relative humidity and at $75^{\circ}\pm 5^{\circ}\text{F}$ for 72 hours minimum. The O-rings shall then be washed with ordinary hand soap and water, rinsed in clear water, and wiped dry.

4.2.4.1 Plates of the metals listed below shall be polished to a surface roughness not to exceed 15 microinches rms finish. The plates shall be washed with solvent naphtha and wiped dry. The metals shall be as follows:

Aluminum alloy; Specification QQ-A-318, Temper H32
Brass; Specification MIL-B-994, Composition A
Bronze; Specification QQ-P-330, Composition A
Steel; Specification MIL-S-6758, Condition C2

4.2.4.2 Within 15 minutes after washing four of the O-rings, they and five metal plates shall be dipped in fluid conforming to MIL-H-19457 and drained to the drip point. The O-rings and plates shall then be so laid together in a stack that at least two O-rings contact each specified metal. The other four O-rings and five of the metal plates shall be dipped in Medium No. 3 fluid and so laid together in a stack that at least two O-rings contact each specified metal. The stacks shall be held together with a force of 20 to 30 pounds, and left in the humidity chamber maintained at 92% minimum relative humidity and at 75 \pm 5 $^{\circ}$ F for 96 hours minimum.

4.2.4.3 When the stacks are disassembled, the tendency of the O-rings to adhere to the metal surfaces shall be noted.

4.2.4.4. The surfaces of the plates which were in contact with the O-rings shall be wiped free of immersion fluid and washed with methyl ethyl ketone. Any pits, eroded marks or deposits remaining after this process, which are visible through a microscope of approximately 10X magnification, shall be construed to be corrosion. Discoloration or staining shall not be considered detrimental.

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Mare Island Naval Shipyard, Rubber Laboratory, Report 92-13 of
29 June 1962

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R. D. Ford and R. E. Morris.

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